



Condensed-Phase Processes During
Solid Propellant Combustion,
V. Further Observations on
Depth Profiling of Burned Surfaces
of XM39 and M43 Propellants

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13. ABSTRACT (Maximum 200 words) <p>Samples of XM39 and M43 propellants that have been self-extinguished at low pressures have been examined under a Scanning Electron Microscope (SEM). The results of this examination suggest that the surface layers of burning HMX/RDX propellants consist of a thin, smooth layer presumably made up of binder and its decomposition products. This layer overlies a thicker layer of crystalline, bubble-containing material that is presumably mostly solidified or recrystallized (liquified during combustion) RDX oxidizer. Photoacoustic Fourier Transform Infrared (FTIR) spectra of some burned/extinguished samples of XM39, JA2, and RDX are also reported and discussed. Based on our results to date, presented in this and previous reports, some suggestions for modelers of nitramine propellant combustion are presented. It is suggested that, if practicable, formation and subsequent decomposition of nitrosoamines, such as MRDX and DRDX, should be included in the condensed-phase chemistry models of deflagration of RDX and of RDX-based propellants. Also, the mathematical framework should include provision for formation and growth of bubbles in the liquid layer as a result of both thermal decomposition and vaporization of RDX.</p>				
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1. INTRODUCTION

This is a progress report on work aimed at understanding the condensed-phase reactions in the combustion of nitramines and other solid gun propellants. Information on the nature and importance of condensed-phase reactions is needed as input for modeling studies. This information could also be very important in understanding the relationship of chemical structure and of physical properties such as melting points, phase transition temperatures, etc., to explosive and propellant behavior.

The authors' initial work has been described in several recent U.S. Army Ballistic Research Laboratory* (BRL) and U.S. Army Research Laboratory (ARL) reports (Schroeder et al. 1990, 1992a, 1992b, 1994). In that work, burning propellant samples were quenched, and the burned surfaces were examined both microscopically and by chemical analysis. Studies (Schroeder et al. 1990, 1992a) were carried out on a series of propellants including XM39, M30, JA2, RDX, and the HMX/binder composition HMX2. Preliminary results on XM39 were consistent with the idea that in combustion of nitramine propellants, at least at low pressures, there is a liquid layer approximately 100–300 μm thick; Scanning Electron Microscope (SEM) examination revealed little, if any, evidence of degradation below this layer. Gas Chromatography Mass Spectroscopy (GCMS) analysis (Schroeder et al. 1990, 1992a) suggests that the stabilizer is depleted considerably in the surface layers, presumably by reaction with nitrogen oxides formed by decomposition of RDX and nitrocellulose (NC). High-Performance Liquid Chromatography (HPLC) results suggest that there is a significant increase in concentration of the mechanistically significant nitrosoamines MRDX and DRDX over the very small amounts possibly present as impurities in RDX. These results are consistent with the idea that RDX decomposition under propellant combustion conditions involves decomposition in the liquid phase, via MRDX and DRDX, to products that are almost entirely gaseous.

Results of SEM examination of the burned surfaces of quenched samples of HMX2, HMX/PU, RDX, M30, and JA2 were also presented (Schroeder et al. 1990, 1992a). Photoacoustic Fourier Transform Infrared (PAS-FTIR) examination (Schroeder et al. 1990, 1992a) of the burned surfaces also provided evidence for condensed-phase reaction in the compositions studied. Some depth-profiling results have also been reported (Schroeder et al. 1994). We have also (Schroeder et al. 1992b) examined the surfaces of some laser-irradiated propellants by SEM and by FTIR microreflectance spectroscopy; by these methods, the laser-irradiated samples appeared similar to burned samples of the same propellants. Our previous reports (Schroeder et al. 1990, 1992a, 1994) contain brief summaries of previous literature on studies of

* The U.S. Army Ballistic Research Laboratory (BRL) was deactivated on 30 September 1992 and subsequently became a part of the U.S. Army Research Laboratory (ARL) on 1 October 1992.

the surface layers of quenched propellant samples of this type. More recently, some very interesting studies (Wilson et al. 1993; Fetherolf et al. 1993) of the surface structure resulting from laser-induced pyrolysis and combustion of M43 and XM39 have been reported.

In the work described here, we have examined a new technique for quenching the combustion of burning propellant grains. A series of propellants has been burned at very low pressures (0.1–0.5 MPa) in a strand burner; at these pressures, the propellant ignites and then burns for a short period of time before self-extinguishing. When XM39 and M43 are burned and extinguished in this way, a cavity or crater is burned into the ignited end of the propellant grain. The quenched surfaces of these samples are free from the disruption and ablation that might be caused by depressurization quenching, or even by quenching with water at atmospheric pressure, and it is much easier to draw conclusions from microscopic examination.

We also report additional FTIR-PAS studies on samples of propellants and RDX that have been burned at atmospheric pressure and quenched by dropping them in water; these experiments provide further information on the variation with depth of chemical composition.

2. EXPERIMENTAL

2.1 Low-Pressure, Self-Extinguished Samples. The unburned propellant grains were unperforated cylinders of 0.5-in outside diameter and approximately 0.5-in length. Their compositions and lot number are given in Table 1. These grains were burned, with hot-wire ignition, in a strand burner under nitrogen, at the pressures indicated in Table 2. Videotapes show that the samples were in fact burning, not just decomposing. At the pressure indicated, the samples burned for a short period of time, then extinguished spontaneously due to the low pressure. At the time of self-extinguishment, the combustion had left a depression in the burned end of the grain, but had not yet spread across the entire end of the grain.

The extinguished grains were cooled with dry ice and split with a knife blade held vertical by mechanical means; when this knife blade was rested against the propellant grain and struck by a hammer, a clean split could be obtained (the knife was mechanically prevented from penetrating more than a small fraction of the grain). One half of the split grain was examined by SEM, and the other half was kept for examination by other methods to be described later.

The electron microscope used was a JEOL Model JSM-820 instrument. Samples were sputtered with gold before examination.

Table 1. Compositions of Propellant Formulations Studied

Propellant (Lot No.)	Composition	Percent
XM39 (IH-XM39-0988-100 A3)	RDX	76.0
	Cellulose Acetate Butyrate (CAB)	12.0
	Acetyl Triethyl Citrate (ATEC)	7.6
	Nitrocellulose (NC) (12.6%N)	4.0
	Ethyl Centralite (EC)	0.4
M43 (IH-HELP-0988-131 B3)	RDX	76.0
	CAB	12.0
	Plasticizer	7.6
	NC (12.6%N)	4.0
	EC	0.4
JA2 (RAD-PE-792-70)	NC (13.04%N)	59.5
	Nitroglycerin (NG)	14.9
	Diethylene Glycol Denitrate	24.8
	EC	0.7
	Magnesium Oxide	0.05
	Graphite	0.05

Table 2. Summary of Samples Studied

Sample No.	Propellant	Pressure (MPa)	Remarks
93100602	M43	0.2	
93100603	M43	0.25	
93100604	M43	0.275	
93100606	M43	0.29	
93100708	XM39	0.25	
93100609	XM39	0.55	Depressurization-Quench

2.2 FTIR-PAS Studies on Extinguished Propellant Samples. Samples of XM39, JA2, and RDX for examination by FTIR-PAS spectroscopy were ignited by a flame, held upright for a few seconds until combustion had stabilized, and then dropped into a beaker of distilled water. After recovery from the water, the samples were pumped out overnight at 0.1 mm, then examined by FTIR-PAS spectroscopy.

FTIR-PAS spectra were obtained on a Mattson Sirius 100 spectrometer using an MTEC 100 photoacoustic cell. The velocity of the interferometer moving mirror was 0.084, 0.115, 0.316, or 0.633 cm/s; the corresponding Fourier frequencies were determined from these values. Sampling depths were then estimated from an existing table (McClelland et al. 1993) of sampling depths vs. thermal diffusivities and frequencies, using the fact that thermal diffusivities for these propellants have recently been measured (Miller 1994a, 1994b) and found to be in the range of 0.001–0.003 cm²/s. All spectra were obtained after thoroughly purging the photoacoustic cell with helium. Spectra were measured at either 4-cm⁻¹ or 2-cm⁻¹ resolution and are the result of 64 co-added scans.

3. RESULTS

A list of samples burned at low pressure with self-extinguishment, together with the pressures at which they were burned, is shown in Table 2. SEM photos of these samples are shown in Figures 1–5.

FTIR-PAS spectra of some burned (air)/quenched (water) and unburned samples of XM39 propellant, JA2 propellant, and military-grade RDX are shown in Figures 6–11.

4. DISCUSSION

4.1 Scanning Electron Microscope (SEM) Studies on XM39 and M43. An overall view of a typical low-pressure self-extinguished nitramine propellant sample is shown in Figure 1. The burned area appears as a hole or crater in the top of the propellant grain. The burned surface generally appears yellow or orange to the eye; dark brown or black areas are also often apparent, especially at the center of the crater and as a ring around the edge of the crater.

SEM examination reveals that the surfaces of these samples contain numerous bubbles, which appear as holes in the surface. The very top layer of the surface usually appears relatively smooth with only occasional signs of crystallization (Figures 2–4), but the layer beneath it, which is 100–300 µm thick (Figures 2 and 4), shows much obvious crystallization, presumably due to the preponderance of RDX in the melt layer. An especially good illustration of this is apparent in Figure 3 (M43) and in Figure 4 (XM39), which show the smooth surface, dotted with bubble-holes, at the bottom of which crystallization is apparent. This is interpreted in terms of the top few micrometers of the melt layer showing relatively few signs of crystallization due to the fact that they contain a large proportion of cellulose acetate



Figure 1. Overall view of typical sample after self-extinguishment.

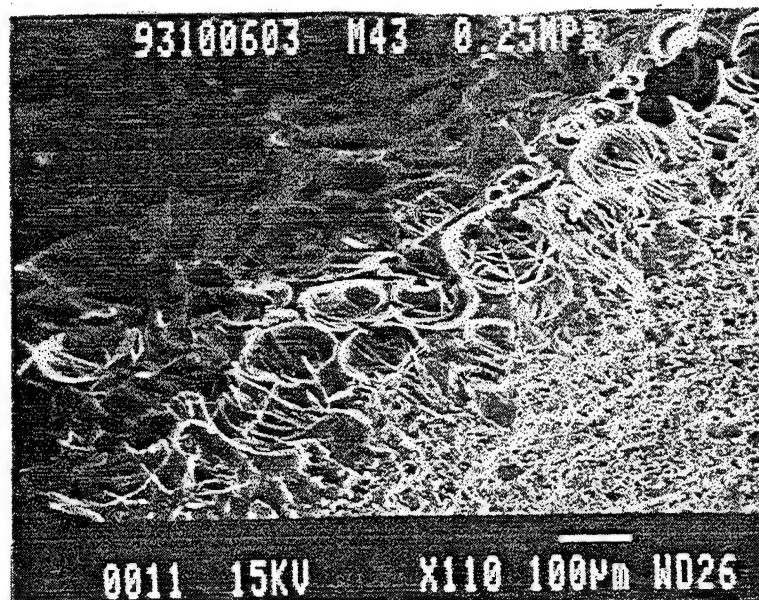


Figure 2. Cross section of burned surface of M43 propellant, self-extinguished at 0.25 MPa.

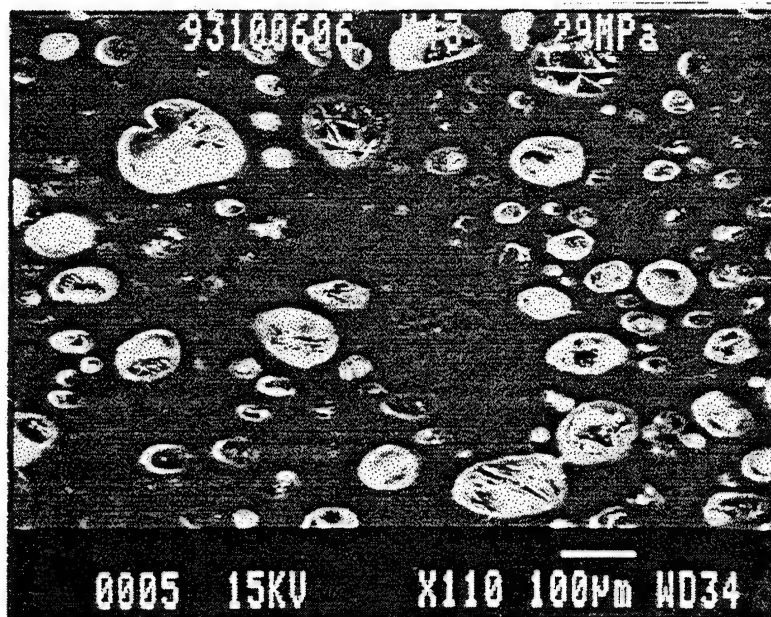


Figure 3. Top of burned surface of M43 propellant, self-extinguished at 0.29 MPa.

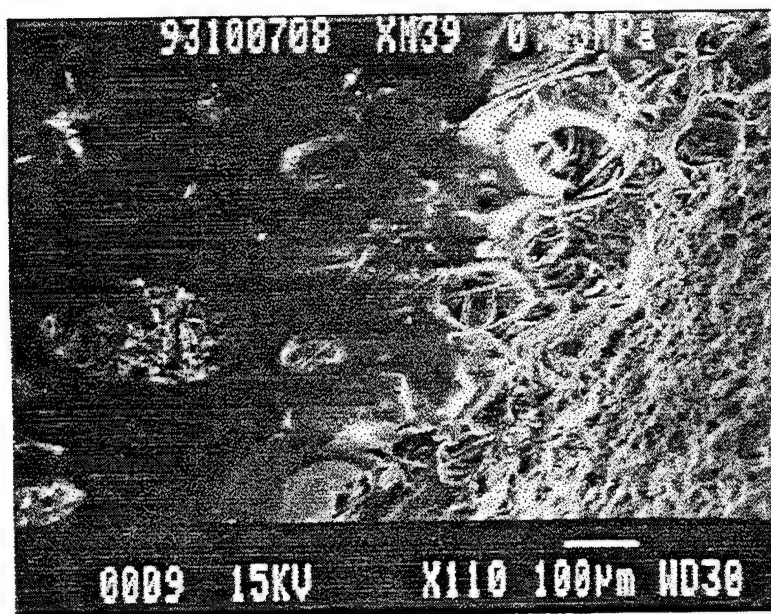


Figure 4. Cross section of burned surface of XM39 propellant, self-extinguished at 0.25 MPa.

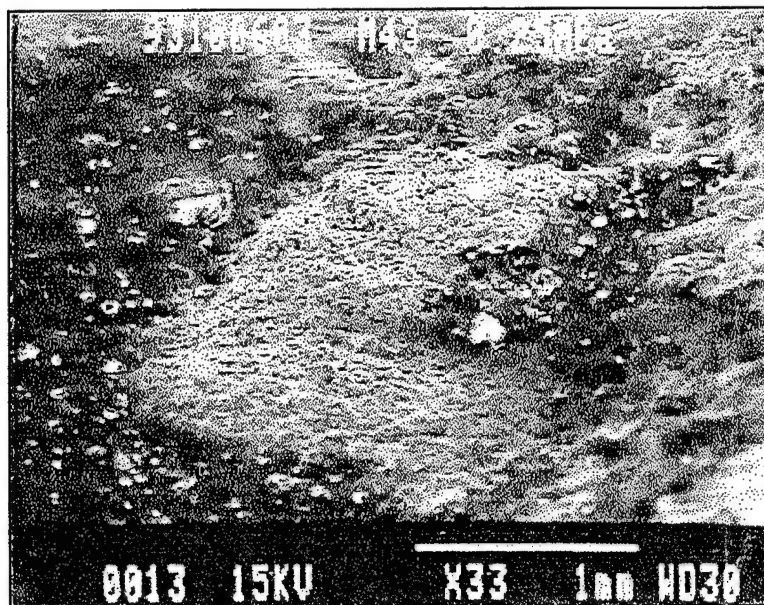


Figure 5. Material overlying burned surface of M43 propellant, self-extinguished at 0.25 MPa.

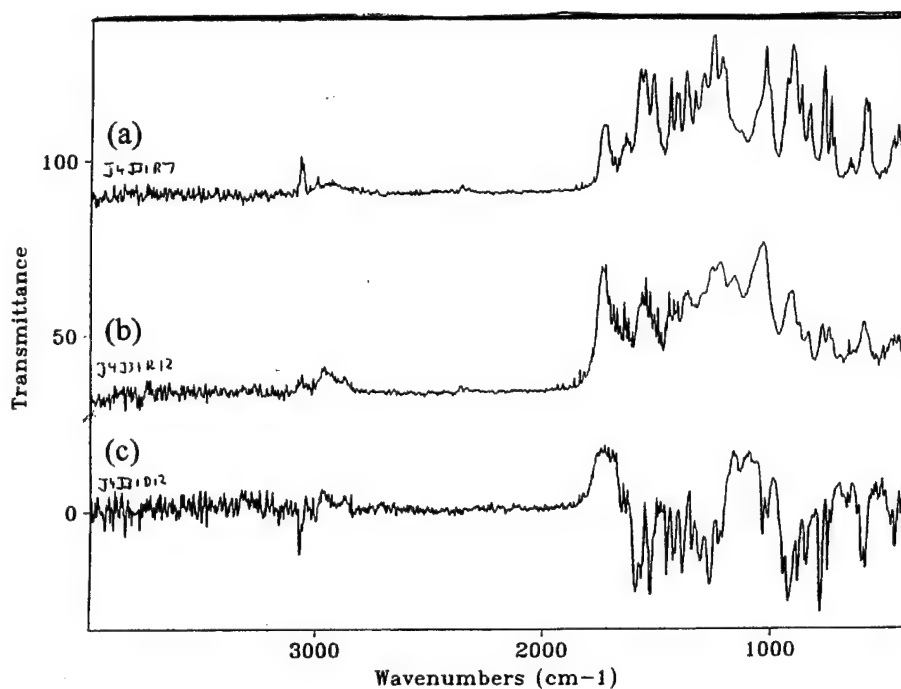


Figure 6. FTIR-PAS spectra of (a) unburned XM39; (b) XM39 burned in air, quenched with water; (c) ratio between (a) and (b). Estimated sampling depth 5–12 μm .

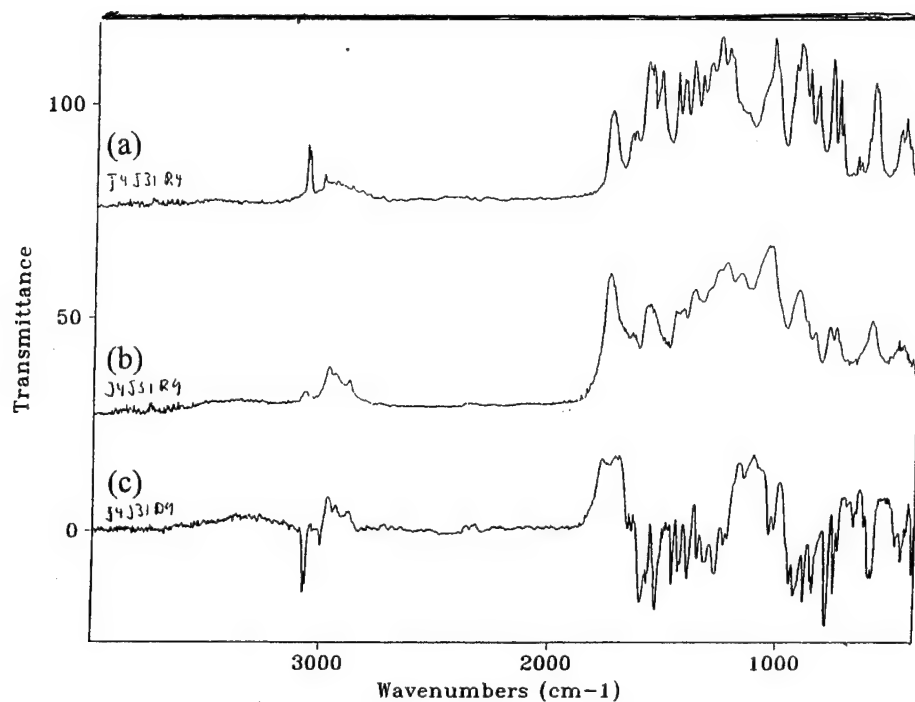


Figure 7. FTIR-PAS spectra of (a) unburned XM39; (b) XM39 burned in air, quenched with water; (c) ratio between (a) and (b). Estimated sampling depth 15–25 μm .

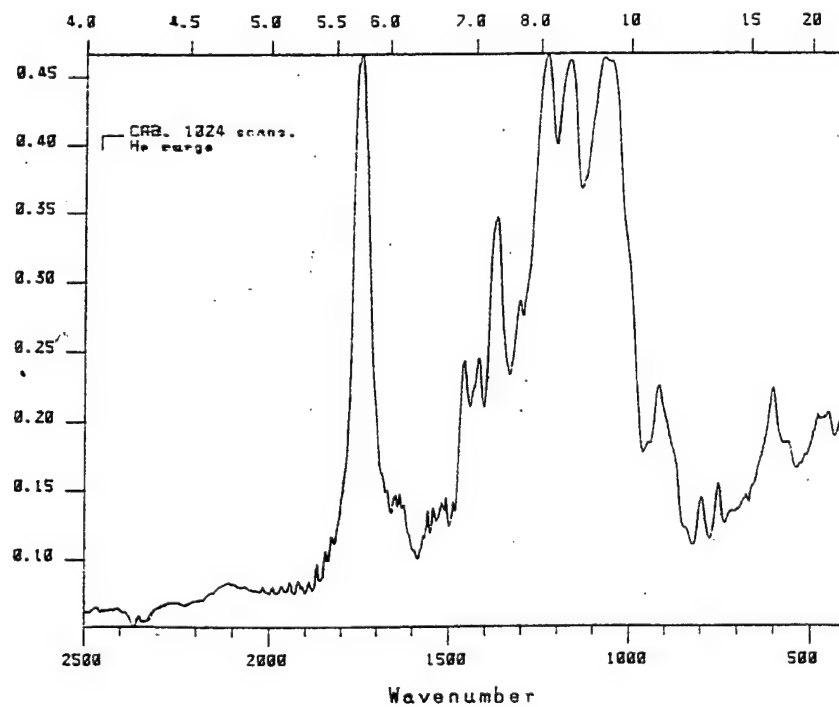


Figure 8. Infrared spectrum of cellulose acetate butyrate (CAB).

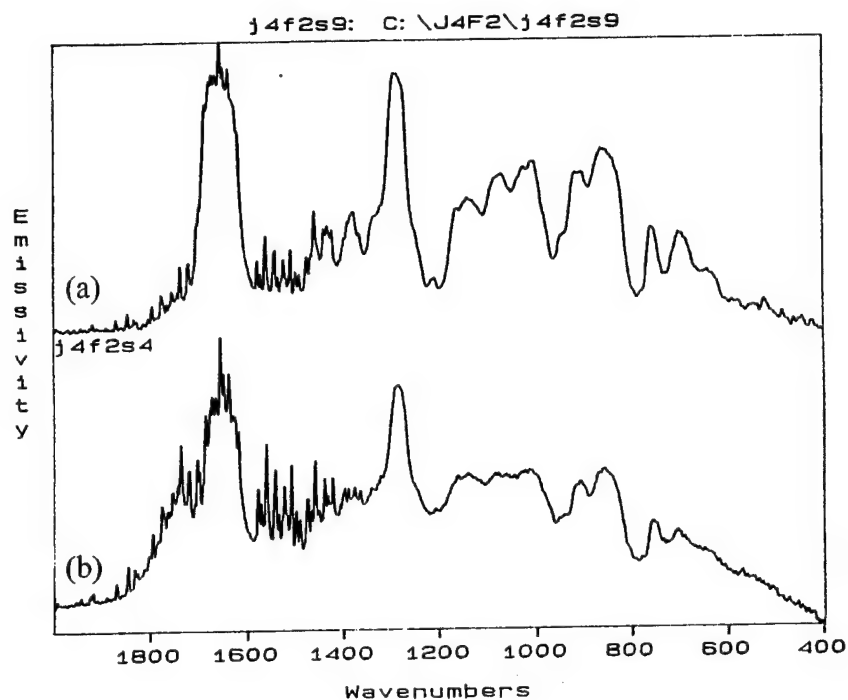


Figure 9. FTIR-PAS spectra of (a) unburned JA2; (b) JA2 burned in air, quenched with water. Estimated sampling depth 5–12 μm .

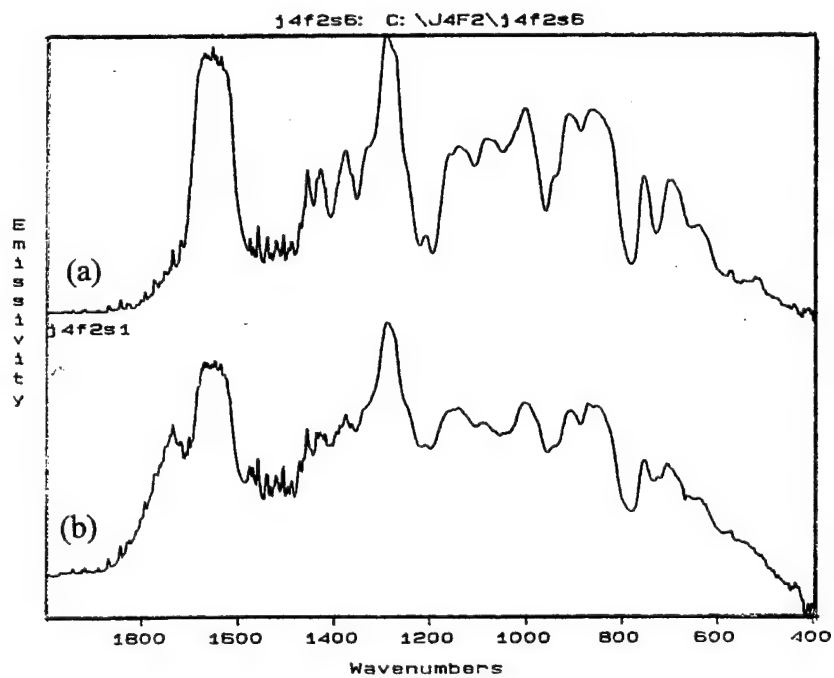


Figure 10. FTIR-PAS spectra of (a) unburned JA2; (b) JA2 burned in air, quenched with water. Estimated sampling depth 15–25 μm .

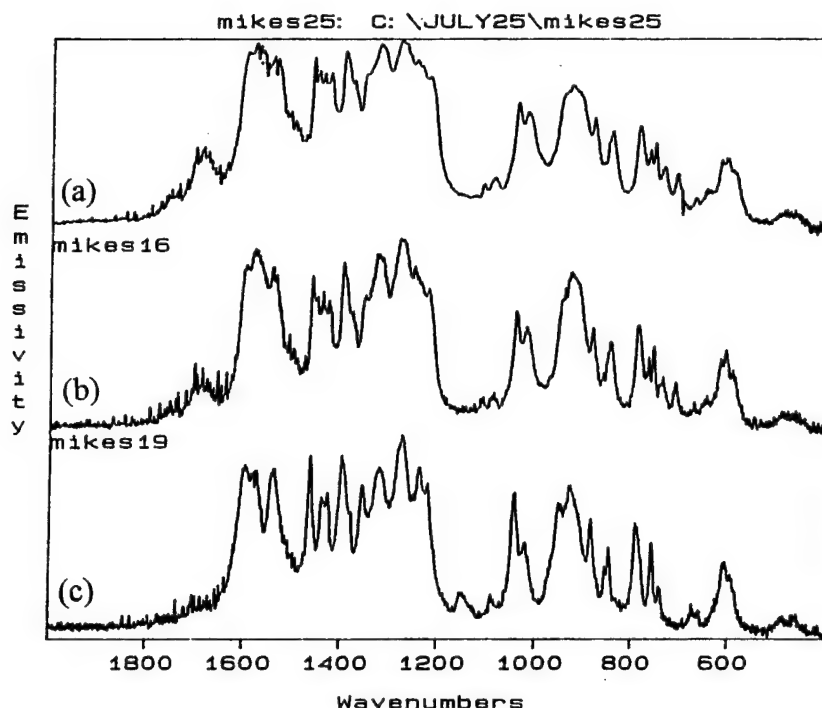


Figure 11. FTIR-PAS spectra of (a) RDX burned in air, quenched with water (estimated sampling depth 15–25 μm); (b) RDX burned in air, quenched with water (estimated sampling depth 5–12 μm); (c) unburned RDX.

butyrate (CAB) and NC binders as well as their polymeric decomposition products. This thin layer appears to be floating on top of a molten layer that is several hundred micrometers thick and contains enough RDX to form crystals readily after self-extinguishment.

On many of the samples, there are patches of something (slag or char?) on the surface of the liquid layer (Figure 5); these patches often seem to correspond to the previously mentioned dark-brown or black areas.

Together with our previous results, these SEM results are consistent with the idea that, at least at low pressures, the burned surfaces of nitramine-binder propellants such as M43 and XM39 consist of a thin layer of binder and decomposition products, with a thicker (up to several hundred micrometers thick) layer composed primarily of molten RDX beneath it.

4.2 FTIR-PAS Studies. In another attempt at depth-profiling, FTIR-PAS experiments have been carried out on propellant samples that were burned in air and subsequently quenched with water. In FTIR-

PAS, the surface of the sample is exposed to infrared (IR) radiation; the sample then experiences oscillatory heating, which results in acoustic waves. These waves can be detected by a microphone. With the help of appropriate computer software, the IR spectra of the near-surface layers of the sample can be extracted from them (McClelland 1993). Using existing tables (McClelland 1993), the sampling depth can be estimated if the modulation frequency and the thermal diffusivity are known. Note that the spectra obtained represent the sum of the entire depth of the sample, from the surface down to the stated depth. Note also the assumption that the thermal diffusivity of the surface (burned) layers is homogeneous and similar to that of the bulk of the sample. Some further assumptions are discussed in the literature (McClelland 1993).

In view of (1) recently measured thermal diffusivities (Miller 1994a, 1994b) for these propellants, and (2) known relationships (McClelland 1993) between thermal diffusivities, instrument modulation frequencies, and sampling depths, the spectra reported here are believed to reflect the composition within about 25 μm of the burned surface; less approximate estimates of sampling depth for specific cases are given in the figure captions and in the following discussion.

4.2.1 FTIR-PAS Studies on Burned/Quenched and on Unburned XM39. Figure 6 shows FTIR-PAS spectra of burned and unburned XM39 propellant, together with the ratio between them; the mirror velocity was such as to result in a sampling depth of about 5–12 μm . Figure 7 shows FTIR-PAS spectra of burned and unburned XM39 propellant, together with the ratio between them. The mirror velocity was such as to result in a sampling depth of about 15–25 μm . As expected, both of these spectra resemble our earlier (Schroeder et al. 1994) FTIR microreflectance (FTIR microscope) spectra of the surface of burned XM39, which seemed to be composed mostly of CAB binder and/or decomposition products with similar IR spectra. This indicates considerable enhancement in CAB binder and decomposition products near the surface. This seems to be in qualitative agreement with our previous conclusion (Schroeder et al. 1994) that, in burning nitramine-binder propellants, the top 10–20 μm of the surface layer are composed of molten binder and decomposition products. Unfortunately, due to the limitations of our equipment, it was impossible to achieve sampling depths greater than about 25 μm .

Figure 8 shows the IR spectrum of CAB; this material is used as binder in XM39. Note that the most intense regions in this spectrum correspond to the most intense regions of the ratio spectra in Figures 6 and 7. This is in agreement with the idea that at these shallow depths, the main constituents are CAB binder and decomposition products having spectra similar to CAB.

4.2.2 FTIR-PAS Studies on Burned/Quenched and on Unburned JA2 Propellant. Figure 9 shows FTIR-PAS spectra of burned and unburned JA2 propellant; the mirror velocity was such as to result in a sampling depth of about 5–12 μm . Figure 10 shows FTIR-PAS spectra of burned and unburned JA2 propellant. The mirror velocity was such as to result in a sampling depth of about 15–25 μm . The main difference between the burned and the unburned samples is the appearance, in the burned sample, of absorption at about $1,735\text{ cm}^{-1}$, characteristics of aliphatic carbonyl-group-containing compounds, such as aldehydes, formed from nitrate-ester decomposition; this is in agreement with our earlier results (Schroeder et al. 1992a, 1994). In our earlier report (Schroeder 1994), it was found that, for nitrate-ester propellants, the burned layer was only about 0–10 μm deep; thus it seems a little surprising that the $1,735\text{-cm}^{-1}$ absorption is not noticeably weaker by the time a depth of 15–25 μm is reached.

4.2.3 FTIR-PAS Studies on Burned/Quenched RDX and on Unburned RDX. Since propellants such as XM39 and M43 contain about 70–80% RDX, it seemed appropriate to get an idea of what changes occur on burning/quenching RDX alone. Figure 11 shows the FTIR-PAS spectra of burned RDX (at two different mirror velocities/sampling depths) and of unburned RDX. The most obvious change caused by burning/quenching is the appearance of absorption bands at $1,750\text{ cm}^{-1}$ and at $1,690\text{ cm}^{-1}$; these are in regions characteristic (Silverstein, Bassler, and Morrill 1991) of carbonyl-group-containing compounds, such as carboxylic acids or esters, and of amides, respectively. Amides have previously been identified among the decomposition products of RDX. These bands increase with increasing sampling depth; possibly this can be explained by one of the following: (a) the bands are associated with a thin layer of decomposition residue which lies on the surface of the burning RDX and which partially decomposes or vaporizes at near-surface depths due to heat from the flame front above the surface; or (b) water-soluble combustion products are being dissolved out of the surface layers of the sample by the water used to quench combustion. In either case, it would appear that some degree of depth-profiling has been effected by the FTIR-PAS approach. The burned/quenched samples also contain additional absorption in the regions around $1,450$, $1,020$, and 710 cm^{-1} . Another interesting point is the appearance, on burning/quenching, of weak absorption in the region around $1,500\text{ cm}^{-1}$; absorption in this region is characteristic of nitrosoamines (Silverstein, Bassler, and Morrill 1991). The appearance of this absorption is thus consistent with our detection of nitrosoamines in burned samples of RDX and XM39 (Schroeder et al. 1990; 1992a). Note also the weak band at $1,150\text{ cm}^{-1}$ in the unburned RDX; the source of this band, which disappears on burning, is unknown.

4.3 Summary and Relationship to Previous Work. At this point, it seems appropriate to briefly summarize our results and conclusions to date, and to discuss their applicability to combustion-modeling efforts, especially those aimed at solid nitramine composite propellants.

First, in the case of nitramine composite propellants such as XM39 and M43 (Figure 12), there appears to be a liquid layer on the burning surface of the propellant. The liquid layer can be seen clearly in SEM photographs in the present work and in our previous work (Schroeder et al. 1990, 1992a). Bubbles, apparently due to decomposition (see following discussion) of RDX are plainly visible, and signs of recrystallization during recovery are evident; the crystallized material is presumably due to the presence of liquid RDX during combustion, followed by crystallization of this liquid RDX on cooling after quenching. The thickness of the liquid varies over the surface, but it is about 100–300 μm thick for samples of XM39 and M43 that have been burned in air at 1 atm, then water-quenched (present work, Schroeder et al. 1990), and somewhat thinner, approximately 100 μm or less, when combustion takes place at pressures of 1.0 MPa and 2.0 MPa under nitrogen (Schroeder et al. 1992a). Very similar results were obtained (Schroeder et al. 1992b) from studies on samples resulting from deradiation-quenching of the combustion of low-pressure, laser-irradiated samples of various propellants. Where comparison is possible, these results agree with recent results from studies on deradiation-quenched samples resulting from low-pressure, laser-assisted combustion of XM39 and M43 (Wilson et al. 1993). These authors also report a number of very interesting relationships between number and size of bubbles, and factors such as pressure and heat flux.

The molten-oxidizer layer is several hundred microns thick and appears to be topped by a thinner layer comprised mostly of molten binder and decomposition products. This follows from several pieces of evidence. First, the FTIR-PAS spectra (Figure 7) (Schroeder et al. 1994) of the recovered (burned/quenched) surfaces of XM39 and other nitramine composite propellants show a considerable increase, over the corresponding spectra of unburned propellant, in the IR absorptions due to the CAB present as binder; presumably this intensity also includes decomposition products of CAB. Second, when the burned surfaces of recovered (burned/quenched) samples are abraded with an abrasive-blaster (Schroeder et al. 1994) and the amount removed is measured, the spectra of the abraded surfaces suggest that, surfaces abraded to estimated depths greater than 10–20 μm below the burned surface contain considerably more RDX than the freshly-quenched, unabraded surface; in fact, their spectra suggest about the same composition as the unburned propellant. (Note that depth was estimated from density and weight of material removed; in view of uneven surface structure and bubbles in the liquid layer, this may be a

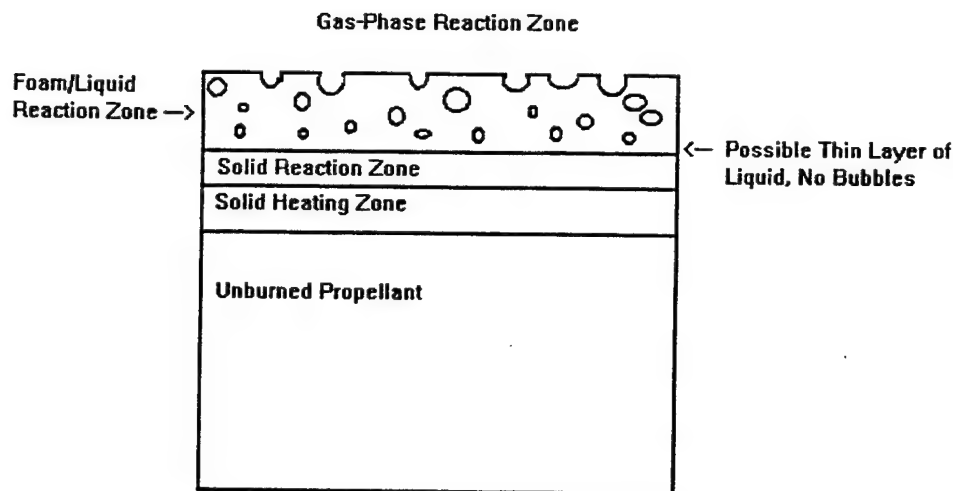


Figure 12. Cross section of burning nitramine propellant.

minimum estimate, but the general trend of more binder/polymeric decomposition products nearer the surface seems valid.) Third (Schroeder et al. 1994), microreflectance-FTIR spectra of the split surfaces of recovered (burned/extinguished) grains of some nitramine composite propellants indicated that, at levels within 20 μm or so of the burned surface, the spectra of the split samples showed elevated levels of absorption bands apparently due to the presence of binder/decomposition products. However at greater depths, the spectra showed mostly nitramine oxidizer, plus smaller amounts of binder. Finally (Section 4.1, Figures 2–4), SEM examination of the burned surfaces and of the cross-sections after splitting of the low-pressure, self-extinguished samples (present work) reveals that the surfaces show only very limited crystallization, while the portions below the burned surface show extensive crystallization. This is attributed to the presence of polymeric binder and decomposition products in the layers closer to the surface, while the material at greater depths into the liquid layer is composed mostly of RDX and so exhibits a much greater degree of recrystallization.

There is also evidence for a significant amount of chemical reaction in the liquid phase ("liquid phase" here refers to the entire liquid layer, from the top of the solid-reaction zone, if any [Figure 12], to the beginning of the gas-phase reaction zone) during the combustion of nitramine propellants. This includes the following observations:

- (a) When grains of XM39 were burned in air and quenched by dropping them into water, or at low pressure with conductive quenching by a block of copper, and the layers affected by the combustion were scraped off and the scrapings analyzed by HPLC (Schroeder et al. 1990, 1992a), it was found that significant amounts (in some cases up to 5–10%, relative to unreacted RDX) of the nitrosoamines MRDX (also referred to as ONDNTA [Behrens and Bulusu 1992]) and DRDX were present in the liquid layer. These nitrosoamines were also found in similar experiments carried out on samples of pure RDX.
- (b) When the scrapings mentioned in (a) were analyzed by GCMS (Schroeder et al. 1990, 1992a), it was found that the amount of stabilizer present (initially 0.4%), had decreased considerably (relative to plasticizer), compared to unburned propellant. In some samples, there was no detectable stabilizer left. The missing stabilizer was presumably destroyed by reaction with nitrogen oxides formed by decomposition of RDX and NC.
- (c) The GCMS and HPLC chromatograms mentioned in (a) and (b) also indicated the presence of small amounts of a number of unidentified compounds; the mass spectra of some of these have been tabulated (Schroeder et al. 1992a). These compounds were presumably formed during combustion.
- (d) It was found in the present work (Figure 11 and Section 4.2.3) that burning of pure RDX in air at atmospheric pressure, followed by quenching in water, led to detection, by FTIR-PAS, of several IR absorptions on the burned surface that are not present in the spectrum of unburned RDX. This constitutes further evidence for occurrence of chemical reaction in the condensed phase.

However, there is evidence that vaporization is also important. Zenin (1995) studied the combustion of HMX and RDX at pressures of 1, 5, 20, and 70 atm by a thermocouple technique and determined a number of properties including (but not limited to) surface temperature, flame temperature, heat release in solid and heat feedback from gas to solid. They found that, for both HMX and RDX, heat release in the solid (or condensed) phase was negative (corresponding to endothermic behavior or heat absorption) at low (1 and 5 atm) pressures, but became positive at higher pressures (20 and 70 atm). This was interpreted by saying that, at low pressures, evaporation (endothermic) made a larger contribution to heat release than did chemical reaction (exothermic), but that at higher pressures the balance shifts and more

heat is released in the condensed phase by chemical reaction than is taken up by evaporation of RDX/HMX. Heat release in the solid phase was defined by the equation

$$Q = c(T_s - T_0) - q - q_r + q_m ,$$

where Q is total heat release in the solid, c is specific heat of the solid, T_s is the surface temperature of the burning propellant, T_0 is the initial temperature, q and q_r are respectively the heat feedback from the gas phase to the burning surface by conduction and radiation, and q_m is the heat of melting. Thus, Q actually refers to the entire condensed (solid and liquid) phase, not just the solid phase. The surface temperatures for RDX at 1 atm and 5 atm were measured as being 320° C and 360° C respectively.

Another estimate of the temperature of the liquid phase can be obtained by the arguments set forth in the following paragraphs. This argument involves the following steps:

- (a) An approximate rate constant for RDX decomposition under combustion conditions is estimated from the burning rate and the melt layer thickness.
- (b) An existing plot (Schroeder 1985b) of rate constant vs. temperature for liquid-phase decomposition of RDX is used to relate the approximate rate constant from (a) to an average temperature at which RDX is disappearing in the liquid layer during combustion.
- (c) The temperature obtained in (b) is then considered to be an approximate estimate of the average temperature in the liquid phase.

We begin by noting that when the copper-quenched RDX and XM39 grains were burned (Schroeder et al. 1992a), burning rates were recorded (Miller 1990). These burn rates varied from about 0.05 cm/s to about 0.2 cm/s, depending on pressure and on the nature of the sample. For this estimation, we use an average value of 0.1 cm/s. Pressures were between 0.25 MPa and 2.0 MPa. For the melt layer thicknesses of these samples, we use an average value of about 70 μ m (Schroeder et al. 1992a). We obtain an approximate value for the rate constant, k , in sec^{-1} , for disappearance of liquid-phase RDX under combustion conditions, by dividing the burning rate (in cm/s) by the thickness of the melt layer (in centimeters); this approximate rate constant is 14 s^{-1} (characteristic time ca 70 μ s). Examination of an

existing (Figure 1 in Schroeder 1985b) plot of $\log_{10} k$ vs. $1,000/T$ indicates that a value of 14 s^{-1} for k corresponds to a value of about 1.7 for $1,000/T$, which yields $T = 588 \text{ K}$ or 315° C . Variations in melt-layer thickness or in burning rate by a factor of 2 cause T to vary between approximately 300° C and 330° C . This range seems to agree satisfactorily with the reported surface temperature of 320° C for RDX burning as a monopropellant at a pressure of 1 atm (Zenin 1995), with a calculated surface temperature of 560 K (287° C) (Liau and Yang 1995) for RDX burning at 1 atm, and with an estimated surface temperature of approximately 620 K (347° C) (Brill 1995) for HMX or RDX burning at 1 atm. Brill (1995) also summarizes several calculated surface temperatures from the literature for HMX and RDX burning at pressures from 0.5 to 100 atm; for RDX at 1 atm, these range from about 549 K (276° C) to 766 K (293° C), with most of them being closer to the lower end of that range. For RDX at 20 atm, a single value of 643 K (370° C) is quoted (Brill 1995).

Note the following approximations and possible sources of error in our previous argument:

- (a) Unlike the pure RDX whose decomposition was used to derive the points in the plot of Figure 1 of Schroeder (1985b), the liquid layer of burning nitramine propellants such as XM39 and M43 undoubtedly contains a significant amount of binder ingredients and their decomposition products.
- (b) The temperature is undoubtedly not constant throughout the melt layer, but is expected to increase from the bottom to the top of the melt layer, and at the top of the melt layer, it is presumably closer to the hypothetical boiling point of RDX.
- (c) Under the rapid-heating conditions characteristic of propellant combustion, it is not inconceivable that, near the top of the melt layer, temperatures may actually reach the boiling point of RDX before the RDX has all disappeared, resulting in boiling in very rapid vaporization of any remaining RDX.
- (d) The RDX may have finished decomposing by the time the outer surface (containing mainly slag and condensed-phase decomposition products) is reached, causing the assumed melt layer thickness to be an upper limit and hence the estimated rate constant and temperature to be lower limits. Note, however, that this effect could be corrected by assuming an artificially small melt layer thickness, and that (preceding paragraph) the effect of variations in assumed melt layer thickness seems to be relatively small.

- (e) The procedure used in this work for estimating the temperature of the melt layer is highly approximate.

Another question that arises is whether the bubbles observed in the liquid layer of burning RDX and RDX propellants are due to thermal decomposition gases, or to the boiling of RDX. Since our results to date (Schroeder et al. 1990, 1992a, 1992b, 1994) indicate that there is reaction in the liquid phase, this question arises regardless of the relative contributions of vaporization and thermal decomposition to the disappearance of RDX from the condensed phase. The available evidence seems consistent with the idea that the bubbles result mainly from thermal decomposition of RDX, rather than from boiling of RDX. This is based on several lines of reasoning.

First, as discussed in the previous paragraphs, RDX is clearly decomposing to some extent in the liquid phase, and this decomposition is expected to evolve bubbles of product gases formed in the decomposition.

Another line of reasoning is based on comparison of the hypothetical boiling point of pure RDX with the temperatures at which decomposition appears to be taking place in the liquid layer of burning RDX. The thermal decomposition of RDX has been studied by many authors (see reviews in Schroeder 1985a and 1985b), including a group at American Cyanamid (Rauch and Fanelli 1969, Rauch and Wainright 1969). This group followed the decomposition by measuring the gases evolved and found the RDX, which melts at 204° C, begins decomposing at higher temperatures. At a temperature of 227° C, increasing scatter in the data was attributed to the rate of decomposition being too rapid ($t_{1/2}$ 75 s) to follow by the manometric procedure employed. The boiling point of pure RDX has never been observed experimentally because of decomposition prior to boiling, but Maksimov (1992) has estimated the hypothetical boiling point of RDX from data on vapor pressure of RDX as a function of temperature; the estimated boiling point for RDX was found to be 391±33° C. Maksimov (1992) also cites an earlier value (Belyaev 1948) of 340° C for the boiling point of RDX, but considers this value unreliable due to the possibility of decomposition of RDX during the measurements. Brill (1995) also reports an estimate of 766 K (493° C) from an application of Trouton's rule to RDX; the accompanying discussion makes it clear that he regards this value and one of 1,097 K (824° C) derived similarly for HMX as being too high, and possibly as evidence of either considerable condensed-phase decomposition under combustion conditions or of variations in heat of evaporation. In the following discussion, we will use the value of Maksimov (1992) (391±33° C) for the boiling point of RDX.

Since decomposition seems to begin at the melting point of RDX and to be proceeding too fast to measure accurately only 20–30° C above this temperature, it seems most reasonable to suppose that, as RDX is heated, it will reach its decomposition point before it reaches the hypothetical boiling point (391° C) of RDX. Furthermore, as was discussed in the preceding paragraphs, the average temperature in the melt layer seems to be in the range of about 300–320° C; this is below the hypothetical boiling point (391° C) of RDX. Based on this information, it seems reasonable to suppose that the bubbles observed in the liquid phase in the present and other (Schroeder et al. 1990, 1992a, 1992b, Fetherolf et al. 1993, Wilson et al. 1993) work on burned/quenched propellant samples are not due primarily to boiling of liquid RDX, but rather are due at least partly to evolution of product gases from thermal decomposition of liquified RDX. However, it seems possible that, especially at temperatures within a few tens of degrees of its melting point, considerable RDX could be entrained in the decomposition gases and carried into the vapor phase.

Third, even if it should turn out that vaporization predominates over decomposition as a pathway for disappearance of RDX from the liquid layer, a small proportion of thermal decomposition relative to vaporization could still account for the observed bubbles. This can be seen by combining the following:

- (a) One mole of an ideal gas occupies a volume of 22.4 liters, and the molecular weight of RDX is 222, and its thermal decomposition is expected to yield six moles of initial products (N_2O , $H_2C=O$, HCN, nitrogen oxides, and acids). If a mole of molten RDX is considered to occupy 222 ml (0.222 liters), its volume is expected to increase on decomposition by a factor of 6 \times (22.4/0.222), or about 600.
- (b) Examination of cross-section photographs of the melt layers of low-pressure burned/quenched samples of RDX and of RDX propellants (Schroeder et al. 1990, 1992a, 1992b; Fetherolf et al. 1993; Wilson et al. 1993) suggests that about one-half of the volume of the melt layer is taken up by the space inside the bubbles. This is consistent with the report from a modeling study (Liau and Yang 1995) that the calculated void fraction was 0.45 at the surface of RDX burning at 1 atm.
- (c) The combination of (a) and (b) suggests that decomposition of as little as $1/(600/0.5) = 8.3 \times 10^{-4}$ of the total RDX present in the melt layer could account for the bubbles present at any one time. Note that this argument is not being made in order to propose that the amount of decomposition

is this small relative to vaporization, but rather in order to point out that even with very small amounts of decomposition relative to vaporization, the bubbles could still be due to the relatively large amounts of gases given off in the decomposition.

As the product gases from decomposition evolve, they presumably assist vaporization of RDX by entraining RDX vapor. This process is probably especially important near the top of the melt layer, where the RDX presumably has a higher vapor pressure as a result of being close to its boiling point. The temperature presumably varies throughout the melt layer, being at the melting point of RDX (ca 200° C) at the lower edge of the melt layer, where there may be a thin, bubble-free layer of liquid RDX. Temperature presumably then increases through the melt until it approaches the hypothetical boiling point of RDX at the top of the melt layer, just before the vapor phase is reached. Presumably, the hypothetical boiling point of RDX (391±33° C) (Maksimov 1992) is an upper limit for temperature in the melt layer. Entrainment of RDX vapor in this way could possibly help vaporization of intact molecules to become a significant process in combustion of RDX and propellants containing RDX.

The preceding discussion is concerned primarily with propellants containing RDX (or HMX) as oxidizer. In the case of other types of propellants examined so far, the condensed-phase-reacted (liquid?) layer seems to be much thinner than for the nitramine propellants, being only about 10 µm thick for M30 (a triple-base propellant containing nitrocellulose [NC], nitroglycerine [NG], and nitroguanidine [NQ]), and less than approximately 5 µm thick for M9 and JA2 (typical nitrate ester propellants made up of NC, with NG [M9] or NG and DEGDN [JA2] as plasticizers). This is based on SEM examination of cross sections of split samples (Schroeder et al. 1992a), and on depth-profiling studies based on the variation in the FTIR-microscope spectra of split or abraded surfaces of burned/quenched samples (Schroeder et al. 1994). These samples were all burned at low pressures (between atmospheric and 2 MPa). These results are in agreement with recent x-ray photoelectron spectroscopic (XPS) studies on the surface layers of burned/quenched nitrate ester propellants (Sharma et al. 1991). Microreflectance-FTIR spectroscopy of the surfaces of our (present work, Schroeder et al. 1994) samples indicates the presence of a peak at approximately 1,730 cm⁻¹, apparently due to carbonyl compounds formed by the thermal decomposition of the nitrate esters in these propellants (e.g., see Fifer 1984).

All of these previously mentioned results seem relevant and of interest to modelers of solid propellant combustion, because they provide an indication of what physical attributes (e.g., melt layer presence and thickness) of a burning sample should be reproduced, at least when combustion is carried out at low

pressures. With regard to combustion of nitramine composite propellants, the results indicate that a liquid layer does exist, and that significant bubble formation and chemical reaction take place in the liquid layer.

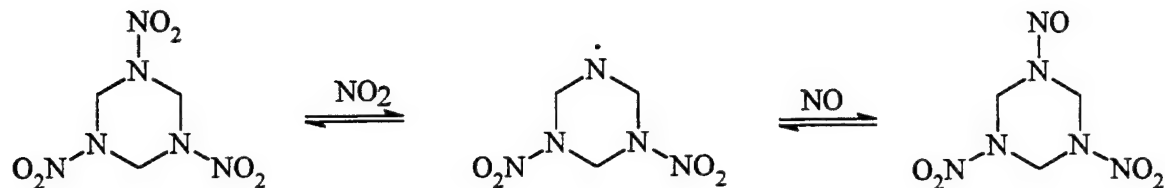
4.4 Suggestions for Modelers. Based on our results in the preceding section, we can offer some suggestions for modelers working in the area of combustion of solid propellants.

As far as we are aware, the most advanced modeling studies currently being carried out on RDX are those being done at The Pennsylvania State University (e.g., see Kuo and Lu 1993; Kuo 1994, 1995; Liau and Yang 1995). The objective of this work is development of a comprehensive theoretical model to describe the detailed reaction zone structure of RDX monopropellant burning under constant ambient pressure. This model includes not only (a) chemical details of the gas-phase flame reactions, but also (b) the solid-phase conductive heating zone (i.e., the solid phase below the liquid or foam layer), and (c) a two-phase (liquid/gas bubble) foam zone, with some degree of detailed chemical representation of gaseous reactions within the bubbles and of reactions within the liquid phase. Note that the thermal decomposition of RDX is a very complex process, and the detailed mechanisms of the reactions involved are in many cases not at all well understood. Furthermore, many of the physical properties of RDX are not well known. Therefore, it would help if we could improve our experience with detailed modeling of three-phase combustion systems of simple, well-known chemistry. Accordingly, Miller (1995) is carrying out modeling studies on the combustion of frozen ozone. This combustion system, like that of RDX, involves gaseous, liquid, and solid phases but has a much simpler chemistry involving a total of only three reversible, elementary reactions for the homogeneous gas phase as well as a single gas-surface reaction. Furthermore, the required physical properties of ozone are better known than those of RDX.

One implication of our work is that, at least in the case of nitramine propellant combustion, there does appear to be some decomposition taking place in the liquid phase; this follows (Schroeder et al. 1990, 1992a) from (a) the observed reduction in stabilizer/plasticizer ratio, and from (b) the detection of nitrosoamines in the surface (liquid) layers. In agreement with this, the modelers mentioned previously (Kuo and Lu 1993; Kuo 1994, 1995; Liau and Yang 1995) are considering two-phase phenomena in the foam layer of their model.

The detection of nitrosoamines in the liquid layer (Schroeder et al. 1990, 1992a) suggests that at least one nitrosoamine-forming reaction should be included in the liquid-layer chemistry. The best candidate

for this reaction is probably a bimolecular combination of NO and the nitrogen-centered radical resulting from loss of a nitro group from RDX:



This is based on the observed formation of the nitrosoamine MRDX (also known as ONDNNTA) with scrambling in "crossover" studies (Behrens and Bulusu 1992) of the decomposition of fully-labeled (with ¹⁵N in both ring and nitro-nitrogens) RDX, and of unlabeled RDX. This reaction could possibly be represented as a typical diffusion-controlled radical recombination reaction; the followup decomposition reactions of MRDX could be represented by analogs of the decomposition reactions included for RDX, replacing N₂O by N₂ and H₂C=NNO₂ by H₂C=NNO where appropriate. We also detected smaller amounts of DRDX (dinitroso analog of RDX); this compound is most likely formed from and sequentially to the mononitroso analog MRDX, by a nitro-nitroso exchange sequence similar to the one mentioned previously. Thus, if the available time and computer facilities allow, it would be interesting to extend the previous approach to include formation of DRDX from MRDX, and possibly even of the trinitroso derivative TRDX from the dinitroso derivative DRDX.

It should be remembered that the liquid/foam layer in combustion of RDX is really a multicomponent system involving a number of components (RDX and its decomposition products) that are or may be gaseous at the temperatures involved, and that any or all of these components may be involved in nucleation processes leading to bubble formation. Therefore, another useful feature of a model would be to include the capability of taking account of simultaneous formation or nucleation of bubbles by both (a) vaporization/boiling involving intact RDX molecules, and by (b) formation from decomposition gases dissolved in the liquid RDX. Possibly the vaporization/boiling component could be based on whatever models may exist for the boiling of more conventional liquids such as water. The component involving formation of bubbles from dissolved decomposition gases could possibly be based on whatever models may exist for processes of applied importance, such as formation, from dissolved carbon dioxide in water, of foam on the top of carbonated beverages or beer. It would also be helpful if the decomposition-

component model included provision for entrainment of RDX vapor in the decomposition-gas bubbles. If formation of bubbles by both (a) and (b) simultaneously were included in the model, the relative amounts of bubbles predicted to form by (a) and by (b) could be compared, leading to improved understanding of the mode(s) of bubble formation.

Finally, combustion modeling will without doubt eventually advance to the point where truly detailed models such as those previously mentioned (Kuo and Lu 1993; Kuo 1994, 1995; Liau and Yang 1995; Miller 1995) can be used to model actual RDX-polymeric-binder propellants rather than just neat RDX as is the case at present. At that time, the significance of phenomena unique to heterogeneous systems (e.g., our finding of higher concentration of molten binder in the upper portions of the surface layers) will become relevant to modelers.

5. WORK NEEDED

Study of the previously listed samples by the methods described is continuing.

Optical microscopic examination of the burned surfaces should be carried out, particularly in view of the possibility that studies of color changes and variations may yield information on the occurrence (or lack thereof) of chemical changes in the solid below the liquid layer.

Additional ways of obtaining improved depth profiling of the burned layers are also being explored; these include microabrasive blasting, microtoming, solvent-dipping, and improved scraping procedures. Depth-profiling will be applied to the nitrosoamine-formation and stabilizer-depletion results reported previously (Schroeder et al. 1990, 1992a). Additional binders, oxidizers, and compositions will be studied. Other propellant formulations will also be examined.

Isotope-scrambling studies on burning (rather than merely decomposing) samples are needed. These would involve use of mixtures of unlabeled RDX or HMX with RDX or HMX labeled with nitrogen-15 in all nitrogens, both in the ring and in the nitro groups. Use of these mixtures would lead to scrambled nitrosoamines if the recombination mechanism were operating, but to unscrambled nitrosoamines if an oxygen-abstraction mechanism were operating. Partial scrambling might mean that both mechanisms were operating to some degree. Note, however, that while formation of fully scrambled nitrosoamines would provide no evidence for oxygen abstraction, it would not necessarily rule it out, since the scrambling could have taken place by further N-N cleavage equilibria before or after formation of the nitrosoamines.

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